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July 1978

Prepared for
U.S. DEPARTMENT OF ENERGY
Office of Energy Technology
Division of Fossil Fuel Utilization



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SUMMARY

Zirconia-based coatings are being evaluated as thermal barriers for air-cooled gas-turbine components. The reactions between partially stabilized zirconia, containing 8-weight-percent yttria, and oxides and sulfates of various elements were studied at 1200⁰, 1300⁰, and 1400⁰ C for times to 800, 400, and 200 hours, respectively. These oxides and sulfates represent impurities and additives potentially present in gas turbine fuels or impurities in the turbine combustion air as well as the elements of the substrate alloys in contact with zirconia. Based on the results, these compounds can be classified in four groups:

- (1) Compounds which did not react with zirconia (Na_2SO_4 , K_2SO_4 , Cr_2O_3 , Al_2O_3 and NiO).
- (2) Compounds that reacted completely with both zirconia phases (CaO , BaO , and BaSO_4).
- (3) Compounds that reacted preferentially with monoclinic zirconia (Na_2O , K_2O , CoO , Fe_2O_3 , MgO , SiO_2 , and ZnO).
- (4) Compounds that reacted preferentially with cubic zirconia (V_2O_5 , P_2O_5).

INTRODUCTION

Many attempts have been made to use refractory oxide coatings as thermal barriers between hot combustion gases and cooled rocket and gas turbine engine parts. The desired role of such coatings was to reduce metal temperature and coolant flow requirements or to increase allowable gas temperatures (refs. 1 to 5). For airfoils, little success had been achieved at temperatures above 1100⁰ C. Recently the two-layer thermal barrier coating developed at NASA's Lewis Research Center (refs. 6 and 7) showed significant improvement over earlier efforts. In the cited NASA work a bond coat (~0.005 to 0.010 cm thick) of Ni-16 Cr - 6 Al - 0.6 Y (weight-percent) was plasma sprayed on superalloy substrates and a thermal barrier coating of ZrO_2 - 12 Y_2O_3 (weight-percent) was similarly applied (~0.025 to 0.064 cm thick).

Because thermal barrier coatings are sought which can protect aircraft or utility gas turbine components at very high temperatures, attractive candidates must withstand thousands of heating-cooling cycles and have a long life without reacting with the impurities contained in the combustion gases or with oxides of the elements in the bond coat and the substrate. The purpose of this investigation was to determine the chemical stability of yttria-stabilized zirconia thermal barrier materials in the presence of such chemical compounds. The reactions were studied at 1200⁰, 1300⁰, and 1400⁰ C for times to 800, 400, and 200 hours, respectively. However, instead of zirconia containing 12 weight-percent yttria, a zirconia with 8 weight-percent yttria content was used throughout this investigation. The reason for this change was unpublished data obtained by S. Stecura of the Lewis Research Center, which revealed that zirconia containing only 8 weight-percent yttria made a longer lived thermal barrier coating.

EXPERIMENTAL PROCEDURE

Materials

Zirconia used in this investigation was in the form of commercially available plasma spray powder (-200 +325 mesh). It's nominal yttria content was 8 weight-percent. The chemical analysis indicated the presence of the following elements in weight-percent: Al-0.005, Ca-0.055, Si-0.042, Fe-0.035, Ni-0.023, Hf-1.70, Na-<0.001, Li-<0.001, K-<0.001, Mg-0.028, Mo-<0.005, Sr-<0.01, Ti-0.027, W-<0.005, Y₂O₃-7.75, and V-<0.001. XRD analysis of the zirconia powder revealed the presence of both the cubic and monoclinic phases. The cubic phase was the predominant one. No free yttria was detected. Heat treatment of this zirconia powder for 100 hours at 1400⁰ did not produce any changes in the relative amounts of the monoclinic and cubic phases. An examination of the polished zirconia particles under the optical microscope (fig. 1(a)) and of the as-received particles by scanning electron microscope (SEM) techniques (fig. 1(b)) disclosed that the particles had

a sponge-like texture. It appears that each particle was a porous cluster of small grains about 0.0003 cm in diameter.

The following chemical compounds, representing impurities in the fuels and in the combustion air as well as the elements of the substrate alloy were reacted with zirconia: Na_2SO_4 , Na_2O , K_2SO_4 , K_2O , CaO , BaSO_4 , BaO , MgO , P_2O_5 , V_2O_5 , Fe_2O_3 , Al_2O_3 , Cr_2O_3 , SiO_2 , NiO , CoO , and ZnO . For practical reasons instead of Na_2O , K_2O , CaO , BaO , and CoO their carbonates were used. Also $(\text{NH}_4)_2\text{HPO}_4$ was substituted for P_2O_5 to facilitate the handling problems. All these compounds were obtained as reagent grade chemicals in powder form. The only exception was SiO_2 , which was procured in the form of a colloidal suspension.

Preparation of the Specimens

Binary mixtures of zirconia and each individual compound were prepared by thoroughly mixing, in a mortar, 1 gram of zirconia with an "equivalent" amount of the compound. The "equivalent" amounts were calculated in the manner that for each atom of Zr there would be an atom of the specific element like Fe, P, Ba, etc. In the case of SiO_2 an appropriate amount of colloidal suspension was added to 1 gram of zirconia. The resulting slurry was dried and ground in a mortar.

All mixtures were packed in small cylindrical containers made of 0.6 cm diameter platinum tubing with one end welded shut. After filling with the powder mixtures the other end was pinched tightly.

Heat Treatment and XRD Analysis

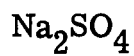
One set of samples so prepared was heated at 1200°C for 400 hours, after which time a small amount of material was removed from each container for XRD analysis. The remainder of each sample in the set was heated at the same temperature for an additional 400 hours. Thus we obtained samples which were heat treated for 400 and for 800 hours.

Similarly, the second set of samples was heated at 1300° C for 200 and 400 hours and the third set was heated at 1400° C for 100 and 200 hours.

After each heat treatment, the samples were ground in a mortar and subjected to XRD analysis. In order to qualitatively evaluate the changes in the relative amounts of cubic and monoclinic zirconia the peak heights of the (111) reflections for both phases were measured and their ratio $R = \text{peak height (111) cubic} / \text{peak height (111) monoclinic}$ was determined when possible. The R value, for as-received zirconia, was approximately 6. During analysis of the obtained data attention was paid not only to the phases which were present but also to the absence of certain phases. Mainly two publications were used as guides in the analysis: Phase Diagrams for Ceramists (ref. 8) and Powder Diffraction File Search Manual (ref. 9).

RESULTS

The results obtained in this investigation are shown below. Each system is discussed separately.



At 1200°, 1300° and 1400° C Na_2SO_4 did not react with zirconia. Both cubic and monoclinic phases were observed before and after such tests. No known compounds of the $\text{ZrO}_2\text{-Na}_2\text{O}$ or $\text{Na}_2\text{O-Y}_2\text{O}_3$ systems were detected. Strong lines of some unidentified compound were detected at all temperatures and times. By leaching one of the specimens with water and analyzing the solution and the residue it was found that the solution did not contain zirconium nor yttrium and the residue was composed of cubic and monoclinic zirconia only. It is reasonable to assume that the unknown compound was a form of sodium sulfate. The absence of any reaction between zirconia and sodium sulfate was also observed by S. J. Dapkunas and R. L. Clarke but at lower temperatures (ref. 5).

Na_2CO_3

In this case sodium carbonate served as a convenient means of introducing Na_2O into the system. After heat treatment at all three temperatures the phases obtained were cubic zirconia, Na_2ZrO_3 and a small amount of an unidentified compound. No monoclinic zirconia was detected. It is reasonable to conclude that Na_2O reacted preferentially with monoclinic zirconia to produce Na_2ZrO_3 .

K_2SO_4

After reacting K_2SO_4 with stabilized ZrO_2 at all three temperatures, the observed phases were: cubic ZrO_2 , monoclinic ZrO_2 and some unknown phase. No K_2SO_4 or known form of potassium zirconate was detected. The relative amount of monoclinic ZrO_2 and of the cubic form containing Y_2O_3 did not vary significantly indicating that potassium sulfate did not react with zirconia. As in the case of sodium sulfate, by leaching one of the specimens and analyzing the solution and residue it was possible to verify that potassium sulfate indeed did not react with cubic nor monoclinic zirconia. The unknown phase must have been some form of unindexed potassium sulfate.

K_2CO_3

After exposure at 1200° and 1300° C the observed phases were monoclinic ZrO_2 and cubic ZrO_2 . A few, very weak diffraction lines could not be indexed. No potassium zirconate, K_2O , KOH or any Y_2O_3 - K_2O compounds were detected. However, the amount of monoclinic ZrO_2 relative to cubic ZrO_2 increased ($R = 2$). After 100 hours at 1400° C the observed phases were cubic ZrO_2 and $\text{K}_2\text{Zr}_3\text{O}_7$. However after 200 hours the only phases noted were cubic and monoclinic zirconia. $\text{K}_2\text{Zr}_3\text{O}_7$ was absent. The behavior of this system could be explained by assuming that K_2O initially reacted with monoclinic ZrO_2 to form $\text{K}_2\text{Zr}_3\text{O}_7$, which upon continued heating, decomposed. In the

process K_2O was lost by evaporation. This interpretation of results is compatible with the work of T. Negas et al. on the influence of K_2O on the cerium oxide- ZrO_2 system (ref. 10).

$CaCO_3$

At all three temperatures the reaction product was $CaZrO_3$ (calcium zirconate). No cubic or monoclinic forms of zirconia were detected.

$BaSO_4$

In the case of $BaSO_4$ (barium sulfate) the experiment was performed only at $1400^{\circ}C$ for 100 and for 200 hours. The observed reaction products were $BaZrO_3$ (barium zirconate) and some unidentified compound. No free barium sulfate nor monoclinic or cubic zirconias were detected.

$BaCO_3$

This was one of the simplest systems to analyze. At elevated temperatures $BaCO_3$ decomposed to CO_2 and BaO , which reacted with zirconia to give $BaZrO_3$. This was the only phase detected after heat treatment at all three temperatures.

MgO

At $1200^{\circ}C$ MgO (magnesium oxide) reacted very slightly with monoclinic zirconia which could be noted from the small increase in the relative amount of cubic zirconia ($R \approx 6.7$). XRD revealed the presence of MgO , cubic ZrO_2 and monoclinic ZrO_2 . At $1300^{\circ}C$ the relative amount of monoclinic ZrO_2 significantly diminished and it could not be detected at all after heat treatments at $1400^{\circ}C$, in which case only cubic ZrO_2 and MgO were found to be present.

SiO₂

The heat treatments at 1200⁰, 1300⁰, and 1400⁰ C produced the following phases: cubic ZrO₂, ZrSiO₄, and SiO₂ (α -cristobalite). Monoclinic zirconia was either not present or it was present only in such small amounts that it was below the XRD detection limits. Another reason for difficulty in detecting monoclinic zirconia was the fact that its diffraction lines coincided with some of those of α -cristobalite and ZrSiO₄. The presence of ZrSiO₄ is the obvious evidence that SiO₂ reacted with zirconia.

(NH₄)₂HPO₄

Because P₂O₅ would be difficult to handle in the normal ambient environment it was necessary to use (NH₄)₂HPO₄ in order to introduce it into the system. After heat treatment at 1200⁰ and 1300⁰ C the following four phases were detected: cubic ZrO₂, monoclinic ZrO₂, ZrP₂O₇, and (ZrO)₂P₂O₇. The relative amounts of the cubic zirconia as well of ZrP₂O₇ were found to decrease with increasing temperature and time. The values of R after 400 and 800 hours at 1200⁰ C were 1.25 and 0.85, and after 200 and 400 hours at 1300⁰ C they were 0.54 and 0.25, respectively. After heat treatment at 1400⁰ C for 100 and 200 hours only monoclinic ZrO₂ and (ZrO)₂P₂O₇ were detected. These results indicated clearly that P₂O₅ reacted preferentially with cubic ZrO₂ to form ZrP₂O₇ which upon continued heating transformed into (ZrO)₂P₂O₇ (which could be written also as 2ZrO₂-P₂O₅) by losing some P₂O₅. The preferential reaction of P₂O₅ with cubic ZrO₂ was recently reported by H. H. Wilson (ref. 11).

V₂O₅

Heat treatments of zirconia-V₂O₅ mixtures at 1200⁰, 1300⁰, and 1400⁰ C produced the same type of results. The detected phases were monoclinic ZrO₂, V₂O₅ and some unidentified compound. No cubic ZrO₂

nor known compounds from the $\text{ZrO}_2\text{-V}_2\text{O}_5$ or the $\text{Y}_2\text{O}_3\text{-V}_2\text{O}_5$ systems were detected. This was a clear indication that V_2O_5 reacted preferentially with the cubic ZrO_2 .



At all three temperatures Fe_2O_3 reacted preferentially with monoclinic ZrO_2 . After heat treatment at 1400°C no monoclinic ZrO_2 was detected, only cubic ZrO_2 and Fe_2O_3 . At 1200° and 1300°C the amount of monoclinic ZrO_2 decreased with reaction time. Because there was no evidence of the existence of other compounds it is concluded that the result of the reaction between Fe_2O_3 and monoclinic ZrO_2 was a cubic ZrO_2 .



No reaction was observed between Cr_2O_3 and zirconia or Y_2O_3 at any temperature studied.



No reaction was observed between Al_2O_3 and zirconia or Y_2O_3 at any temperature studied.



No reaction was observed between NiO and zirconia or Y_2O_3 at any temperature studied.



At 1400°C CoCO_3 decomposed to CO_2 and CoO , which in turn reacted with monoclinic ZrO_2 to form cubic ZrO_2 . The only phases de-

tected by XRD were cubic ZrO_2 and CoO . No experiments were run at 1200° or 1300° C.

ZnO

At 1200° and 1300° C ZnO reacted preferentially with monoclinic ZrO_2 . The observed phases were cubic ZrO_2 , monoclinic ZrO_2 and some unidentified compound. No ZnO was detected. After heat treatment at 1400° C all monoclinic zirconia was gone and so was ZnO . Only cubic ZrO_2 and the unknown compound were detected. It is reasonable to assume that this compound was a result of reaction between monoclinic ZrO_2 and ZnO .

The above described results are summarized in table I. This table lists all the chemical compounds used in this investigation and products of their reactions with zirconia at 1200° , 1300° , and 1400° C. It also includes a column marked with letters N or Y indicating that no reaction took place or that yes, there was a partial or complete reaction.

DISCUSSION

Based on the obtained results, the chemical compounds which were used to react with zirconia can be divided into four distinct groups:

Group 1. Chemical compounds that did not react with zirconia:

Na_2SO_4 , K_2SO_4 , Cr_2O_3 , Al_2O_3 , and NiO

Group 2. Chemical compounds that reacted completely with zirconia:

CaCO_3 (CaO), BaCO_3 (BaO), and BaSO_4

Group 3. Chemical compounds that reacted preferentially with monoclinic ZrO_2 :

Na_2CO_3 (Na_2O), K_2CO_3 (K_2O), CoCO_3 (CoO), Fe_2O_3 , MgO , SiO_2 , and ZnO

Group 4. Chemical compounds that reacted preferentially with cubic ZrO_2 :

V_2O_5 , $(\text{NH}_4)_2\text{HPO}_4$ (P_2O_5)

Although in practical use zirconia thermal barriers will operate in an environment much more complex than the conditions under which these experiments were performed, the information obtained from this investigation should prove to be valuable not only in the analysis of the problems one could encounter during testing or use of zirconia thermal barriers in such environments but this information could also be useful in developing new improved barriers. For example, it might not be advisable to use CoCrAlY or FeCrAlY type alloys as the bond coat material because of the reactivity between CoO and Fe_2O_3 and monoclinic zirconia. Also, the use of zirconia thermal barriers in an environment containing particles of iron oxide (near steel mills and foundries) would not be indicated for the same reason. Also, fuel additives containing barium should be avoided because both monoclinic and cubic zirconia react readily with BaO and BaSO_4 to form barium zirconate. It is very significant that neither Na_2SO_4 nor K_2SO_4 reacted with monoclinic and cubic zirconia. These are a few simple examples of the applicability of the information obtained in this investigation.

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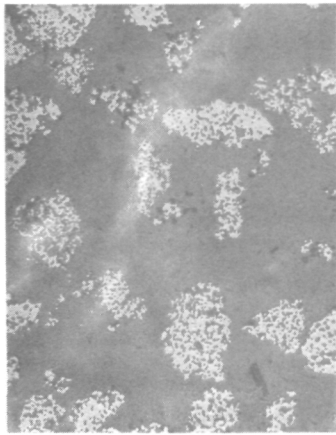
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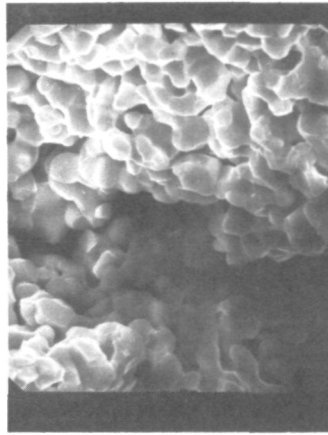
TABLE I. - SUMMARY OF REACTIONS BETWEEN YTTRIA STABILIZED ZIRCONIA WITH OXIDES AND SULFATES OF VARIOUS ELEMENTS

Temperature	1200° C				1300° C				1400° C			
Time	400 hr		800 hr		200 hr		400 hr		100 hr		200 hr	
Na ₂ SO ₄	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N ^c	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N
Na ₂ CO ₃ ^a (Na ₂ O)	ZrO ₂ cubic Na ₂ ZrO ₃	Y ^d	ZrO ₂ cubic Na ₂ ZrO ₃	Y	ZrO ₂ cubic Na ₂ ZrO ₃	Y	ZrO ₂ cubic Na ₂ ZrO ₃	Y	ZrO ₂ cubic Na ₂ ZrO ₃	Y	ZrO ₂ cubic Na ₂ ZrO ₃	Y
K ₂ SO ₄	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N	ZrO ₂ cubic ZrO ₂ monoclinic Unknown phase	N
K ₂ CO ₃ ^a (K ₂ O)	ZrO ₂ cubic ZrO ₂ monoclinic	Y	ZrO ₂ cubic ZrO ₂ monoclinic	Y	ZrO ₂ cubic ZrO ₂ monoclinic	Y	ZrO ₂ cubic ZrO ₂ monoclinic	Y	ZrO ₂ cubic K ₂ Zr ₃ O ₇	Y	ZrO ₂ cubic ZrO ₂ monoclinic	Y
BaSO ₄	Not tested		Not tested		Not tested		Not tested		BaZrO ₃ Unknown phase		BaZrO ₃ Unknown phase	
BaCO ₃ ^a (BaO)	BaZrO ₃	Y	BaZrO ₃	Y	BaZrO ₃	Y	BaZrO ₃	Y	BaZrO ₃	Y	BaZrO ₃	Y
CaCO ₃ ^a (CaO)	CaZrO ₃	Y	CaZrO ₃	Y	CaZrO ₃	Y	CaZrO ₃	Y	CaZrO ₃	Y	CaZrO ₃	Y
MgO	ZrO ₂ cubic ZrO ₂ monoclinic MgO	N	ZrO ₂ cubic ZrO ₂ monoclinic MgO	N	ZrO ₂ cubic ZrO ₂ monoclinic MgO	Y	ZrO ₂ cubic MgO Trace of monoclinic ZrO ₂	Y	ZrO ₂ cubic MgO	Y	ZrO ₂ cubic MgO	Y
SiO ₂	ZrO ₂ cubic ZrO ₂ monoclinic SiO ₂ ^e ZrSiO ₄	Y	ZrO ₂ cubic SiO ₂ ZrSiO ₄ Trace of monoclinic ZrO ₂	Y	ZrO ₂ cubic ZrO ₂ monoclinic SiO ₂ ZrSiO ₄	Y	ZrO ₂ cubic SiO ₂ ZrSiO ₄	Y	ZrO ₂ cubic SiO ₂ ZrSiO ₄	Y	ZrO ₂ SiO ₂ ZrSiO ₄	Y
(NH ₄) ₂ HPO ₄ ^b (P ₂ O ₅)	ZrO ₂ monoclinic ZrO ₂ cubic ZrP ₂ O ₇ (ZrO) ₂ P ₂ O ₇	Y	ZrO monoclinic ZrO ₂ cubic ZrP ₂ O ₇ (ZrO) ₂ P ₂ O ₇	Y	ZrO ₂ monoclinic ZrO ₂ cubic ZrP ₂ O ₇ (ZrO) ₂ P ₂ O ₇	Y	ZrO ₂ monoclinic ZrO ₂ cubic ZrP ₂ O ₇ (ZrO) ₂ P ₂ O ₇	Y	ZrO ₂ monoclinic (ZrO) ₂ P ₂ O ₇	Y	ZrO ₂ monoclinic (ZrO) ₂ P ₂ O ₇	Y
V ₂ O ₅	ZrO ₂ monoclinic V ₂ O ₅ Unknown phase	Y	ZrO ₂ monoclinic V ₂ O ₅ Unknown phase	Y	ZrO ₂ monoclinic V ₂ O ₅ Unknown phase	Y	ZrO ₂ monoclinic V ₂ O ₅ Unknown phase	Y	ZrO ₂ monoclinic V ₂ O ₅ Unknown phase	Y	ZrO ₂ monoclinic V ₂ O ₅ Unknown phase	Y
Fe ₂ O ₃	ZrO ₂ cubic ZrO ₂ monoclinic Fe ₂ O ₃	N	ZrO ₂ cubic Fe ₂ O ₃ Trace of monoclinic ZrO ₂	Y	ZrO ₂ cubic ZrO ₂ monoclinic Fe ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Fe ₂ O ₃	Y	ZrO ₂ cubic Fe ₂ O ₃	Y	ZrO ₂ cubic Fe ₂ O ₃	Y
Cr ₂ O ₃	ZrO ₂ cubic ZrO ₂ monoclinic Cr ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Cr ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Cr ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Cr ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Cr ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Cr ₂ O ₃	N
Al ₂ O ₃	ZrO ₂ cubic ZrO ₂ monoclinic Al ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Al ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Al ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Al ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Al ₂ O ₃	N	ZrO ₂ cubic ZrO ₂ monoclinic Al ₂ O ₃	N
NiO	ZrO ₂ cubic ZrO ₂ monoclinic NiO	N	ZrO ₂ cubic ZrO ₂ monoclinic NiO	N	ZrO ₂ cubic ZrO ₂ monoclinic NiO	N	ZrO ₂ cubic ZrO ₂ monoclinic NiO	N	ZrO ₂ cubic ZrO ₂ monoclinic NiO	N	ZrO ₂ cubic ZrO ₂ monoclinic NiO	N
CoCO ₃ ^a (CoO)	Not tested		Not tested		Not tested		Not tested		ZrO ₂ cubic CoO	Y	ZrO ₂ cubic CoO	Y
ZnO	ZrO ₂ cubic Unknown phase	Y	ZrO ₂ cubic Unknown phase	Y	ZrO ₂ cubic Unknown phase	Y	ZrO ₂ cubic Unknown phase	Y	ZrO ₂ cubic Unknown phase	Y	ZrO ₂ cubic Unknown phase	Y

^aZrO₂ was reacted with the oxides indicated in the parentheses. Carbonates were the convenient form for handling these oxides.^bP₂O₅ was introduced in the form of (NH₄)₂HPO₄.^cN indicates that no reaction occurred.^dY indicates a partial or complete reaction.^eα-cristobalite.



(a) As polished cross section.
No etch, X250.



(b) SEM, X3000.

Figure 1. - Microstructure of zirconia spray powder containing 8 wt % Yttria.

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16. Abstract Zirconia coatings are being evaluated as thermal barriers for air-cooled gas-turbine components. The reactions of Y_2O_3 - stabilized zirconia with various oxides and sulfates were studied at 1200 ⁰ , 1300 ⁰ , and 1400 ⁰ C for times to 800, 400, and 200 hours, respectively. These compounds represent potential impurities or additives in gas turbine fuels and in turbine combustion air as well as elements of the substrate alloys in contact with the zirconia. The phase compositions of the reaction products were determined by x-ray diffraction analysis.			
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